PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 : C13D 3/14, B01D 15/08 — A1

(11) International Publication Number: WO 94/17213

(43) International Publication Date: 4 August 1994 (04.08.94)

(21) International Application Number: PCT/FI94/00024

(22) International Filing Date: 21 January 1994 (21.01.94)

(30) Priority Data:
930321 26 January 1993 (26.01.93) FI

(71) Applicant *(for all designated States except US)*: SUOMEN SOKERI OY [FI/FI]; Kyllikinportti 2, FIN-00240 Helsinki (FI).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: HEIKKILÄ, Heikki [FI/FI]; Aallonkohina 4 C 27, FIN-02320 Espoo (FI). HYÖKY, Göran [FI/FI]; Linkoojanrinne 7, FIN-02460 Kantvik (FI). KUISMA, Jarmo [FI/FI]; Edis I.E. 38, FIN-02460 Kantvik (FI).

(74) Agent: OY KOLSTER AB; Iso Roobertinkatu 23, P.O. Box 148, FIN-00120 Helsinki (FI).

(81) Designated States: AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, LV, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, US, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: A METHOD FOR THE FRACTIONATION OF MOLASSES

(57) Abstract

The invention relates to a method for the fractionation of molasses using a chromatographic simulated moving bed system in which the liquid flow is effected in a single direction in a system comprising at least two chromatographic partial packing material beds. In the method of the invention, the product or products are recovered during a multi-step sequence comprising the following phases: feeding phase of molasses, eluting phase and recycling phase. The liquid present in the partial packing material beds with their dry solids profile is recycled in the recycling phase in a loop comprising one, two or several partial packing material beds.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	**GB**	United Kingdom	**MR**	Mauritania
AU	Australia	**GE**	Georgia	**MW**	Malawi
BB	Barbados	**GN**	Guinea	**NE**	Niger
BE	Belgium	**GR**	Greece	**NL**	Netherlands
BF	Burkina Faso	**HU**	Hungary	**NO**	Norway
BG	Bulgaria	**IE**	Ireland	**NZ**	New Zealand
BJ	Benin	**IT**	Italy	**PL**	Poland
BR	Brazil	**JP**	Japan	**PT**	Portugal
BY	Belarus	**KE**	Kenya	**RO**	Romania
CA	Canada	**KG**	Kyrgystan	**RU**	Russian Federation
CF	Central African Republic	**KP**	Democratic People's Republic of Korea	**SD**	Sudan
CG	Congo			**SE**	Sweden
CH	Switzerland	**KR**	Republic of Korea	**SI**	Slovenia
CI	Côte d'Ivoire	**KZ**	Kazakhstan	**SK**	Slovakia
CM	Cameroon	**LI**	Liechtenstein	**SN**	Senegal
CN	China	**LK**	Sri Lanka	**TD**	Chad
CS	Czechoslovakia	**LU**	Luxembourg	**TG**	Togo
CZ	Czech Republic	**LV**	Latvia	**TJ**	Tajikistan
DE	Germany	**MC**	Monaco	**TT**	Trinidad and Tobago
DK	Denmark	**MD**	Republic of Moldova	**UA**	Ukraine
ES	Spain	**MG**	Madagascar	**US**	United States of America
FI	Finland	**ML**	Mali	**UZ**	Uzbekistan
FR	France	**MN**	Mongolia	**VN**	Viet Nam
GA	Gabon				

A method for the fractionation of molasses

The present invention relates to a method for the fractionation of molasses using a chromatographic simulated moving bed system in which the liquid flow is effected in a single direction in a system comprising at least two chromatographic partial packing material beds. Fractionation of molasses denotes fractionation of various vegetable-derived by-products of the food and fermenting industries, such as beet and cane molasses, stillage, vinasse, slop, wood molasses, corn steep water, wheat, barley and corn molasses (hydrolyzed C starch). In the method of the invention, the product or products are recovered during a multi-step sequence comprising the following phases: feeding phase of molasses, eluting phase and recycling phase.

The liquid present in the partial packing material beds with their dry solids profile is recycled in the recycling phase in a loop comprising one, two or several partial packing material beds.

These phases are employed to form sequences comprising several process steps. In accordance with the invention, a sequence preferably comprises five to ten steps. A step comprises for example

a molasses feeding phase and/or feeding of an eluant liquid and recovery of the product or products, or

an eluting phase with recovery of a product or products, or

recycling phase and eluting phase with recovery of a product or products, or

two or more recycling phases.

Sequences comprising said steps are repeated five to seven times to reach an equilibrium.

Typically from three to twelve, preferably three

to six chromatographic partial packing material beds are employed. A loop may comprise one, two or several partial packing material beds. Strongly acid cation exchange resin is preferably used as the column packing material.

The simulated moving bed system has been developed and introduced by UOP (United Oil Products), U.S.A., at the beginning of the 1960's, initially for petrochemical applications (U.S. Patent 2,985,589). Today several simulated moving bed methods for a number of different applications are known (U.S. Patents 3,706,812, 4,157,267, 4,267,054, 4,293,346, 4,312,678, 4,313,015, 4,332,623, 4,359,430, 4,379,751, 4,402,832, 4,412,866, 4,461,649, 4,533,398 and 5,127,957, and published European application 0,279,946).

The simulated moving bed system enables separating performances that are many times higher, and lower dilution of the products (consumption of eluant) than in the batch method.

The simulated moving bed method is either continuous or sequential.

In a continuous simulated moving bed method, all flows are continuous. These flows are: feeding of feed solution and eluant liquid, recycling of liquid mixture and recovery of products (usually only two). The flow rate for these flows may be adjusted in accordance with the separation goals (yield, purity, capacity). Normally, 8 to 20 partial packing material beds are combined into a single loop. In accordance with the above-mentioned U.S. Patent 4,402,832, the recycling phases have been applied to the recycling of dilute fractions. The feed and product recovery points are shifted cyclically in the downstream direction. On account of the feed of eluant liquid and feed solution (and on account of recovery of products) and the flow through the packing

material bed, a dry solids profile is formed. Ingredients having a lower migration rate in the packing bed are concentrated at the downstream end of the dry solids profile, and respectively ingredients having a higher migration rate at the upstream end. Feeding points for feeding solution and eluant liquid and recovery points for product or products are shifted gradually at substantially the same rate at which the dry solids profile moves in the bed. The product or products are recovered substantially from the upstream and downstream end of the dry solids profile, and the feed solution is fed approximately to the maximum point of the dry solids profile and the eluant liquid approximately to the minimum point of the dry solids profile. Part of the separated product fraction is recycled on account of the continuous cyclic flow and as only part of the dry solids profile is removed from the packing material bed.

The cyclical shifting of the feed and recovery points is performed by using feed and recovery valves disposed along the packing material bed at the upstream and downstream end of each partial packing material bed. If it is desired to recover product fractions of high purity, short phase times and a plurality of partial packing material beds must be employed (the apparatus has corresponding valves and feed and recovery equipment).

In a sequential simulated moving bed method, not all flows are continuous. In a sequential simulated moving bed method the flows are: feeding of feed solution and eluant liquid, recycling of liquid mixture and recovery of products (two to four or more products; e.g. betaine as a third fraction in beet molasses separation and monosaccharides in cane sugar separation). The flow rate and the volumes of the different feeds and product fractions may be adjusted in accordance with the separ-

ation goals (yield, purity, capacity). The method comprises three basic phases: feeding, elution, and recycling. During the feed phase, a feed solution and possibly also an eluant liquid is fed into predetermined partial packing material beds, and simultaneously two or even three product fractions are recovered. During the eluting phase, eluant liquid is fed into a predetermined partial packing material bed, and during said phases one or even two product fractions are recovered in addition to the residue. During the recycling phase, no feed solution or eluant liquid is fed into the partial packing material beds and no products are recovered.

Finnish Patent Application 882740 (U.S. Patent 5,127,957) discloses a method for recovery of betaine and sucrose from beet molasses using a sequential simulated moving bed method, the chromatographic system therein comprising at least three chromatographic partial packing material beds connected in series and adapted for the flow of liquids in a single direction in partial packing material beds, in which method betaine and sucrose are separated during the same sequence comprising:

- molasses feeding phase, in which a molasses feed solution is fed into one of said partial packing material beds and in which eluant water is fed substantially simultaneously into another partial packing material bed,
- feeding phase of eluant water, and
- recycling phase,

these phases being repeated either once or several times during the sequence.

A novel sequential simulated moving bed method has now been developed, which is particularly suitable for the fractionation of molasses. The novel method yields a purer sucrose solution with a better yield and/or capa-

city.

In the novel method, the liquid flow is arranged in a single direction in a system comprising at least two partial packing material beds, and the product is/products are recovered during a multi-step sequence. The partial packing material bed usually comprises one column. The sequence comprises feeding, eluting and recycling phases. During the recycling phase, the liquid in the partial packing material beds with their dry solids profile is recycled in a loop comprising one, two or several partial packing material beds.

Therefore, in the novel method recycling is employed in a novel way. In the recycling phase one, two or three or even more separate successive loops are formed. For example, the number of columns being four, the loop preferably comprises two columns. The loop may be closed or "open", i.e., when liquid is recycled in the other loop, eluant liquid can be fed into the other loop and the product fraction can be recovered therefrom. During feed and elution, the flow through the packing material beds may be effected between successive loops, the flows conveying material from one loop to another. During the recycling phase, the loop is closed and separated from the other loops. One separate dry solids profile is recycled in each loop.

Molasses is rich in various coloured components (colourants) which were difficult to remove completely enough by the earlier methods. Separate colour-removing phases were needed, or two-step crystallization had to be used to obtain a colourless product. The novel method according to the invention affords even 90% or greater colour removal in the molasses fractionating phase alone. The major part of the colour is separated already in the column group of that loop to which the feed solution is supplied, and it will not essentially contaminate the

column groups of the second (or third) loop. Continuous and stable colour removal is achieved. When a batch method or conventional simulated moving bed methods (continuous or sequential), for instance, are employed, colour removal is normally only 75-80% in continuous long-term separation.

Also the separation of non-sugars, i.e. salts, is efficient when the method of the invention is used, and thus the sucrose content of the sugar fraction obtained from the separation can be very high, usually in advantageous cases in excess of 92-95% on the dry solids. The majority of the salts is separated already in the column group of that loop to which the feed solution is supplied, and thus the ion exclusion is more complete in the following loops. The result is a more symmetrical, sharper and higher sucrose peak, in other words, better separation of sucrose is achieved. When the bath method or conventional simulated moving bed methods (continuous or sequential), for instance, are employed, the sucrose content of the sugar fraction is in advantageous cases usually about or below 90-92% on the dry solids.

A strongly acid, gel-type cation exchange resin (e.g. "Zerolit 225", "Finex" or "Purolite") preferably in the sodium or potassium form is used as a packing for the columns.

Prior to the chromatographic fractionation, the feed solution (beet molasses) is preferably diluted with water to 20-65% by weight, softened with sodium carbonate and finally filtered using diatomaceous earth as a filtering aid. Prior to feed into separation columns, the molasses solution is heated to 40-85°C and even to 95°C.

Water preferably at 40-85°C is used for the elution.

The flow rate of the liquid in the columns is 0.5-

10 $m^3/h/m^2$, even 20 $m^3/h/m^2$.

The following examples illustrate the novel sequential simulated moving bed method for the fractionation of molasses. These examples shall not be regarded as restricting the scope of the invention, as they are only examples of employing the method according to the invention to recover sucrose and betaine from beet molasses.

Example 1

A pilot plant scale chromatographic test apparatus was employed. The apparatus included four columns, feed pumps, recycling pumps, eluant water pumps, flow and pressure regulators, and inlet and outlet valves for the different process streams. The flowchart is shown in Figure 1.

The columns were packed with a strongly acid cation exchange resin ("Purolite"). The resin had a polystyrene/divinylbenzene backbone and was activated with sulphonic acid groups; the mean spherule size was about 0.36 mm. The resin had a DVB content of 5.5%. Initially the resin had been regenerated to sodium form, and during the run it was balanced with cations from the feed molasses.

Test conditions:

Diameter of columns	200 mm
Height of resin bed/ column	2800 mm
Temperature	75°C
Flow rate	40, 50, 70 and 90 l/h

The feed solution consisted of beet molasses wherefrom calcium had been removed by adding sodium carbonate (pH 9) and filtering the precipitate off using diatomaceous earth as an aid.

The separation of sucrose and betaine was performed by an eight-step sequence in which each column had its

specific function. As shown in Figure 1, steps 5, 6 and 7 each comprise one recycling phase and one feeding phase for eluant water, and step 8 two recycling phases. The duration of the sequence was 79 minutes and the sucrose yield 84.0% (on the amount of sucrose fed).

Step 1: Molasses was fed (feeding phase) into column 1 at flow rate 50 l/h, and the residue fraction was eluted from the downstream end of the column. Simultaneously water was supplied (eluting phase) to column 2 at a flow rate 90 l/h, and a recycling fraction and sucrose were eluted from column 4. Said recycling fraction was used to dilute the raw material (molasses).

Step 2: Feeding of molasses into column 1 and elution of residue from the downstream end of column 1 were continued. Simultaneously water was supplied to columns 2 and 4 at a flow rate 90 l/h, the residue fraction was eluted from column 3, and the elution of sucrose was continued from column 4.

Step 3: Water was fed into columns 1 (50 l/h) and 4 (90 l/h), and the residue fraction was eluted from columns 1 and 3.

Step 4: Water was fed into column 2 at a rate 90 l/h, and the residue fraction was eluted from column 3.

Step 5: Recycling (recycling phase) in columns 1 and 2 at a rate 90 l/h; simultaneously water was supplied to column 3 at a rate 70 l/h and the betaine fraction was eluted from column 4.

Step 6: Water was fed into column 1 at a rate 90 l/h and the residue fraction was eluted from column 2; simultaneous recycling in columns 3 and 4 at a rate 70 l/h.

Step 7: Recycling in columns 1 and 2 at a rate 90 l/h.

Step 8: Recycling in columns 1 and 2 at a rate 90

l/h and in columns 3 and 4 at a rate 40 l/h.

After the sequence was completed, the process control program was continued and it returned to step 1. By repeating this sequence five to seven times, an equilibrium was reached in the system. The run was continued in a state of equilibrium, and product fractions with a constant composition were recovered and analyzed (cf. Tables 1 and 2).

The progress of the separation process was monitored with a density meter, a meter for optical activity, and a conductivity meter, and the separation was controlled by a microprocessor whereby precisely defined volumes and flow rates of feeds, recycled liquid and product fractions were controlled employing quantity/volume measuring, valves and pumps.

Table 1 shows the volumes of the feeds, recycled liquid and product fractions, and Table 2 shows the compositions of molasses and the product fractions. The sucrose and betaine fractions were recovered from column 4. Table 5 shows the colours of the molasses, residues and product fractions.

Table 1

Volumes of feeds, recycled liquid and product fractions (1)

Step No.	1	2	3	4	5	6	7	8
Molasses feed	18[x)]		-	-	-	-	-	-
Water feed	21	5.0+8.5	4.0+8.0	5.0	26.0	25.0	0	-
Raffinate fraction from column 1	18[x)]		4.0	-	-	-	-	-
Raffinate fraction from column 2, 3 or 4	-	5.0	8.0	5.0	-	25.0	0	-
Betaine fraction	-	-	-	-	26.0[xx)]	-	-	
Recycle fraction	7.5	-	-	-	-	-	-	-
Sucrose fraction	13.5	8.5	-	-	-	-	-	-
Recycled solution	-	-	-	-	26.0	20.0	26.0[xxx)]	

[x)] Total from steps 1 and 2

[xx)] Total from steps 4 and 5

[xxx)] Total from steps 7 and 8

Table 2

Compositions of feed and product fractions

	Dry solids (kg/l)	Sucrose (% by weight on d.s.)	Betaine (% by weight on d.s.)	Other substances (% by weight on d.s.)
Molasses feed	0.76	58.2	5.6	36.2
Residue fraction	0.075	21.2	7.5	71.3
Betaine fraction	0.028	10.1	41.4	48.5
Sucrose fraction	0.279	94.8	0.7	4.5

Example 2

A pilot plant scale chromatographic test apparatus was employed. The apparatus included three columns, feed pumps, recycling pumps, eluant water pumps, flow and pressure regulators, and inlet and outlet valves for the different process streams. The flowchart is shown in Figure 2.

The columns had been packed with a strongly acid cation exchange resin ("Purolite"). The resin had a polystyrene/divinylbenzene backbone and was activated with sulphonic acid groups; the mean spherule size was about 0.36 mm. The resin had a DVB content of 5.5%. Initially the resin had been regenerated to sodium form, and during the run it was balanced with cations from the feed molasses.

Test conditions:

Diameter of columns	200 mm
Height of resin bed:	
columns 1 and 3	4100 mm
column 2	2800 mm
Temperature	75°C
Flow rates	25, 35, 45, 85 and 110 l/h

The feed solution consisted of beet molasses

wherefrom calcium had been removed by adding sodium carbonate (pH 9) and filtering the precipitate off using diatomaceous earth as an aid.

The separation of sucrose and betaine was performed by a five-step sequence in which each column had its specific function. As shown in Figure 2, steps 2 and 3 each comprise one recycling phase and one feeding phase for eluant water, and step 5 two recycling phases. The duration of the sequence was 100 minutes and the sucrose yield 87.3% (on the amount of sucrose fed).

Step 1: Molasses was fed into column 1 at flow rate 45 l/h, and residue was eluted from the same column (downstream end of the column); simultaneously water was supplied to column 2, and a recycling fraction and sucrose fraction were eluted from column 3 at a flow rate 85 l/h.

Step 2: Water was fed into column 2 at a rate 110 l/h, and the residue fraction was eluted from column 1; simultaneous recycling in column 3 at a rate 25 l/h.

Step 3: Recycling in columns 1 and 2 at a rate 110 l/h; simultaneously water was supplied to column 3 at a rate 35 l/h and the betaine fraction was eluted from the same column.

Step 4: Water was fed into column 1 at a rate 110 l/h and into column 3 at a rate 35 l/h, and the residue fraction was eluted from columns 2 and 3.

Step 5: Recycling in columns 1 and 2 at a rate 110 l/h and in column 3 at a rate 25 l/h.

After the sequence was completed, the process control program was continued and it returned to step 1. By repeating this sequence five to seven times, an equilibrium was reached in the system. The run was continued in a state of equilibrium, and product fractions with a constant composition were recovered and analyzed.

Table 3 shows the volumes of the feeds, recycled

solution and product fractions, and Table 4 shows the compositions of molasses and the product fractions. Table 5 shows the colours of the molasses, residues and product fractions.

Table 3

Volumes of feeds, recycled liquid and product fractions (l)

Step No.	1	2	3	4	5
Molasses feed	18	-	-	-	-
Water feed	33.3	5.0	13.0	34.0+10.0	-
Residue fraction from column 1	18	5.0	-	-	-
Residue fraction from column 2 or 3	-	-	-	34.0+10.0	-
Betaine fraction	-	-	13.0	-	-
Recycle fraction	7.3	-	-	-	-
Sucrose fraction	26.0	-	-	-	-
Recycled solution	-	6.0	26.0	-	44.0+5.0

Table 4

Compositions of feed and product fractions

	Dry solids (kg/l)	Sucrose (% by weight on d.s.)	Betaine (% by weight on d.s.)	Other substances (% by weight on d.s.)
Molasses feed	0.760	57.1	5.4	37.5
Residue fraction	0.069	18.7	6.8	74.5
Betaine fraction	0.048	5.3	47.5	47.2
Sucrose fraction	0.264	89.4	1.0	9.6

Table 5

Colours of molasses and product fractions

Example 1

	colour ICUMSA	residue 1	residue 2	residue 3
Molasses	47700			
Residue	115400	123600	151000	43324
Betaine	29900			
Sucrose	2100			

Example 2

	colour ICUMSA	residue 1	residue 2	residue 3
Molasses	38250			
Residue	92500	136000	240600	25900
Betaine	21800			
Sucrose	4300			

Example 3

A pilot plant scale chromatographic test apparatus was employed. The apparatus included three columns, feed pumps, recycling pumps, eluant water pumps, flow and pressure regulators, and inlet and outlet valves for the different process streams. The flowchart is shown in Figure 3.

The columns were packed with a strongly acid cation exchange resin ("Finex"). The resin had a polystyrene/divinylbenzene backbone and was activated with sulphonic acid groups; the mean spherule size was about 0.36 mm. The resin had a DVB content of 5.5%. Initially the resin had been regenerated to sodium form, and during the run it was balanced with cations from the feed molasses.

Test conditions:

Diameter of column	200 mm
Height of resin bed:	
columns 1, 2 and 3	5000 mm
Temperature	75°C
Flow rates	22, 35, 40, 45, 70, 75 l/h

The feed solution consisted of beet molasses wherefrom calcium had been removed by adding sodium carbonate (pH 9) and filtering the precipitate off using diatomaceous earth as an aid.

The separation of sucrose and betaine was performed by a five-step sequence in which each column had its specific function. As shown in Figure 3, step 3 comprises one recycling phase and step 5 three recycling phases. The duration of the sequence was 111 minutes and the sucrose yield 81.9% (on the amount of sucrose fed).

Step 1: Molasses was fed into column 1 at a flow rate 35 l/h, and the recycling fraction and sucrose fraction were eluted from column 3.

Step 2: Water was fed into column 1 at a flow rate 70 l/h and the sucrose and recycling fractions were eluted from column 3.

Step 3: Recycling in column 1 at a flow rate 70 l/h; simultaneously water was supplied to column 2 at a flow rate 40 l/h and the betaine fraction was eluted from column 3.

Step 4: Water was fed into columns 1, 2 and 3 at flow rates 70, 75 and 40 l/h, the residue fractions were eluted from columns 1, 2 and 3, and the elution of the betaine fraction was continues from column 3.

Step 5: Recycling in columns 1, 2 and 3 at flow rates 22, 75 and 45 l/h.

After the sequence was completed, the process control program was continued and it returned to step 1. By repeating this sequence five to seven times, an

equilibrium was reached in the system. The run was continued in a state of equilibrium, and product fractions with a constant composition were recovered and analyzed.

Table 6 shows the volumes of the feeds, recycled solutions and product fractions, and Table 7 shows the compositions of the molasses and product fractions.

Table 6

Volumes of feeds, recycled liquid and product fractions (l)

Step No.	1	2	3	4	5
Molasses feed	20				
Water feed		20	10	20+26+20	
Residue fraction					
from column 1				26	
from column 2				26	
from column 3				15	
Betaine fraction			10	5	
Recycle fraction	9	4			
Sucrose fraction	11	16			
Recycled solution			8		11+12+11

Table 7

Compositions of feed and product fractions

	Dry solids (kg/l)	Sucrose (% by weight on d.s.)	Betaine (% by weight on d.s.)	Other substances (% by weight on d.s.)
Molasses feed	0.754	59.6	5.6	34.8
Residue fraction	0.081	16.7	8.8	74.5
Betaine fraction	0.071	45.9	22.9	31.2
Sucrose fraction	0.252	92.7	0.7	6.6

Claims:

1. A method for the fractionation of molasses using a chromatographic simulated moving bed system in which the liquid flow is effected in a single direction in a system comprising at least two chromatographic partial packing material beds, characterized in that the product or products are recovered during a multi-step sequence comprising the following phases: feeding phase of molasses, eluting phase and recycling phase, wherein the liquid present in the partial packing material beds with their dry solids profile is recycled in the recycling phase in a loop comprising one, two or several partial packing material beds.

2. A method as claimed in claim 1, characterized in that each loop comprises one whole dry solids profile.

3. A method as claimed in claims 1 and 2, characterized in that a step comprises a molasses feeding phase and/or one or more feeding phases of an eluant liquid, and a recovery phase of a product or products.

4. A method as claimed in claims 1 and 2, characterized in that a step comprises one or more recycling phases, and a feeding phase for eluant liquid and a product recovery phase.

5. A method as claimed in claims 1 to 4, characterized in that the product or products comprise a residue and sucrose and/or betaine.

6. A method as claimed in claims 1 and 2, characterized in that a step comprises two or more recycling phases.

7. A method as claimed in claims 1 and 2, characterized in that a sequence comprises five to ten steps.

8. A method as claimed in any one of claims 1 to 7, characterized in that a sequence comprising said steps is repeated five to seven times in order to reach an equilibrium in the system, and the method is continued with in the state of equilibrium reached.

9. A method as claimed in any one of claims 1 to 8, characterized in that a system comprising three to twelve, preferably three to six chromatographic partial packing material beds is employed.

10. A method as claimed in any one of claims 1 to 9, characterized in that a partial packing material bed is one column.

11. A method as claimed in any one of claims 1 to 10, characterized in that a strongly acid cation exchange resin is employed as a packing material for said columns.

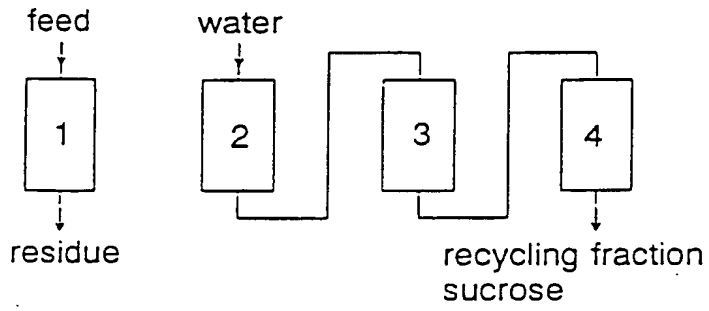
12. A method as claimed in claim 11, characterized in that said strongly acid cation exchange resin is in monovalent form, preferably in sodium or potassium form, or as a mixture of these forms.

13. A method as claimed in any one of claims 1 to 12, characterized in that the flow rate of the liquid in the columns is 0.5-10 $m^3/h/m^2$, even 20 $m^3/h/m^2$.

14. A method as claimed in any one of claims 1 to 13, characterized in that the temperature of the feed solution and eluant water is about 40-85°C, even 95°C.

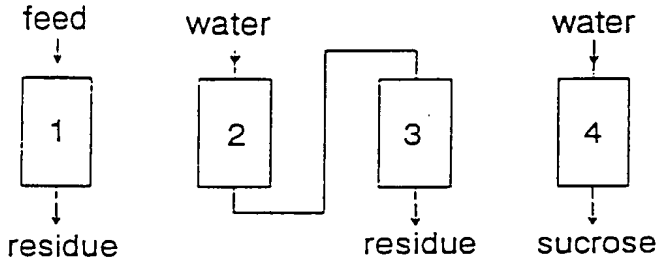
15. A method as claimed in any one of claims 1 to 14, characterized in that the dry solids content of the feed solution is 20-65% by weight, even 80% by weight.

FIG. 1



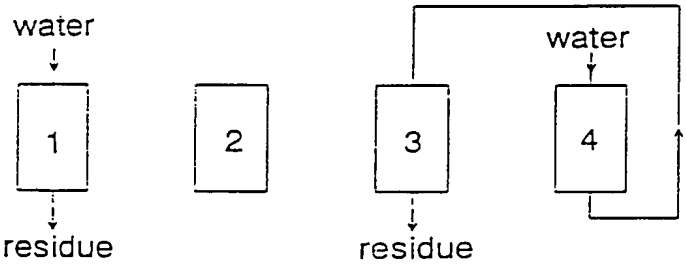
STEP 1

feed	18 l	(50 l/h)
residue	18 l	(50 l/h)
recycling fraction	7.5 l	(90 l/h)
sucrose	13.5 l	(90 l/h)



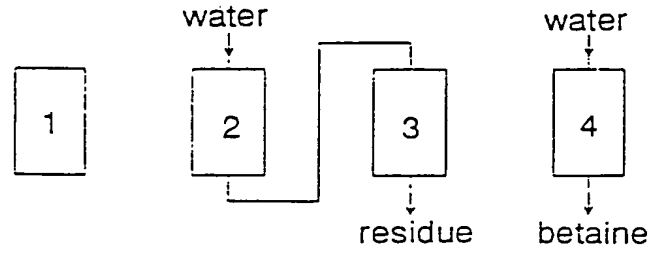
STEP 2

residue (1) (50 l/h)		(cont'd)
residue (3) (90 l/h)	5.0 l	
sucrose (90 l/h)	8.5 l	



STEP 3

residue (1)	4.0 l	(50 l/h)
residue (3)	8.0 l	(90 l/h)



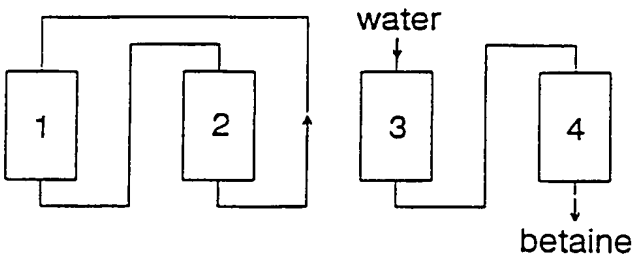
STEP 4

residue	5.0 l	(90 l/h)
betaine	0 l	

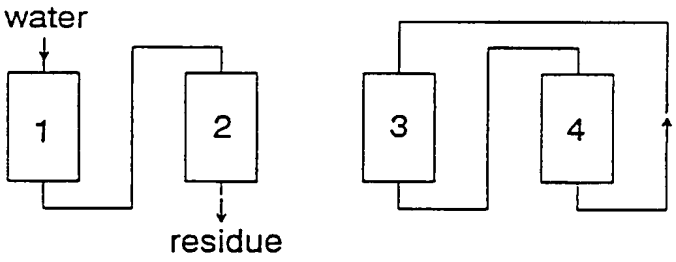
cont'd

FIG. 1 (cont'd)

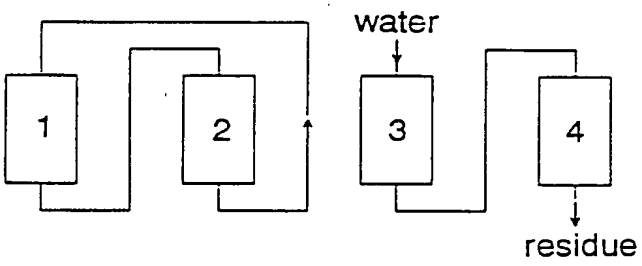
Loop I Loop II



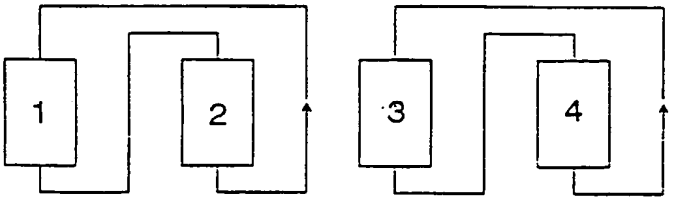
STEP 5
recycl. (loop I) 26 l
(90 l/h)
betaine 26 l
(70 l/h)



STEP 6
residue 25 l
(90 l/h)
recycl. (loop II) 20 l
(70 l/h)

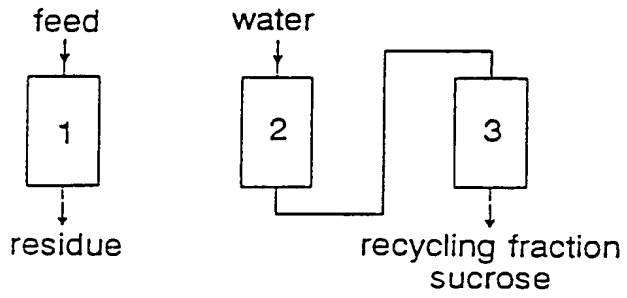


STEP 7
recycl. (loop I) 26 l
(90 l/h)
residue 0 l



STEP 8
recycl. (loop I) cont'd
(90 l/h)
recycl. (loop II) 6 l
(40 l/h)

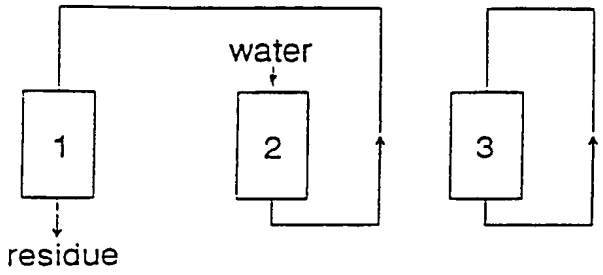
FIG. 2



STEP 1

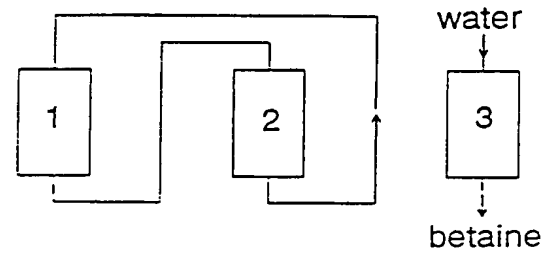
feed	18 l	(45 l/h)
residue	18 l	(45 l/h)
recycling fraction	7.3 l	(85 l/h)
sucrose	26 l	(85 l/h)

Loop I Loop II



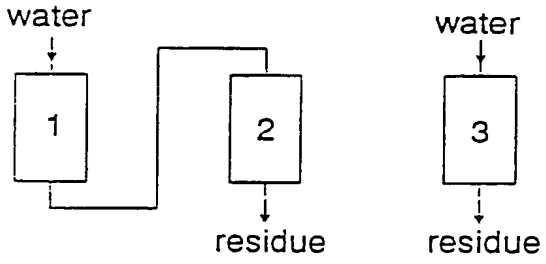
STEP 2

residue (loop I) 5 l (110 l/h)
recycl. (loop II) 6 l (25 l/h)



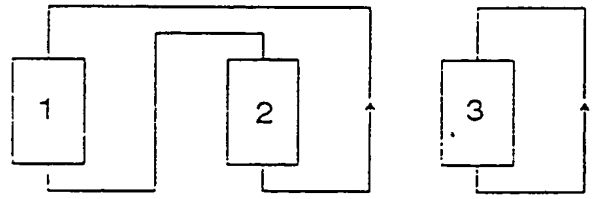
STEP 3

recycl. (loop I) 26 l (110 l/h)
betaine 13 l (35 l/h)



STEP 4

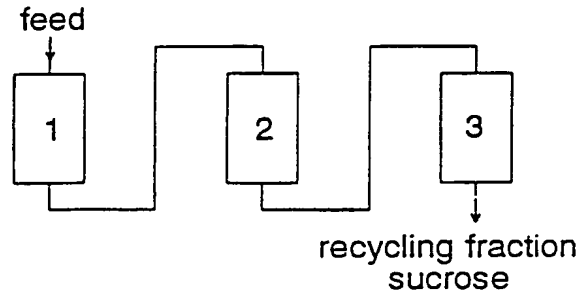
residue (loop I) 34 l (110 l/h)
residue (loop II) 10 l



STEP 5

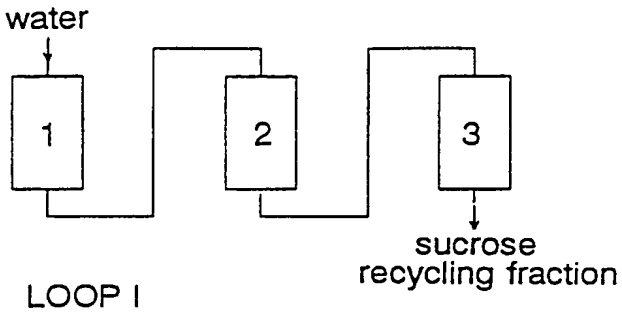
recycl. (loop I) 44 l (110 l/h)
recycl. (loop II) 5 l (25 l/h)

FIG. 3



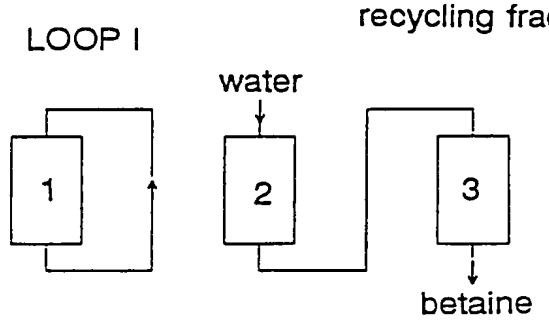
STEP 1

feed	20 l	(35 l/h)
recycling fraction	9 l	(35 l/h)
sucrose	11 l	(35 l/h)



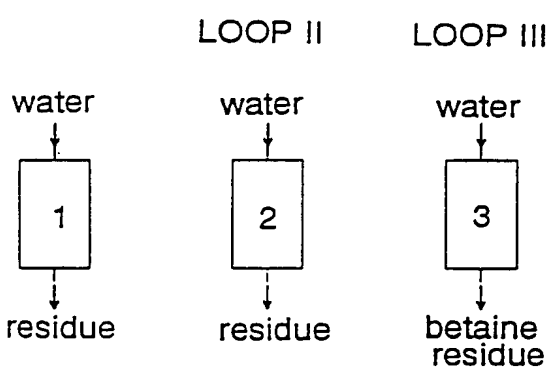
STEP 2

sucrose	16 l	(70 l/h)
recycling fraction	4 l	(70 l/h)



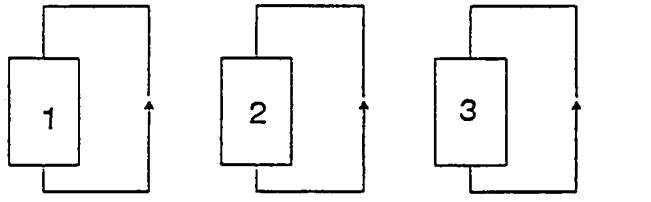
STEP 3

recycl. (loop I) (70 l/h)	8 l
betaine (40 l/h)	10 l



STEP 4

1. residue	26 l	(70 l/h)
2. residue	26 l	(75 l/h)
betaine	5 l	(40 l/h)
3. residue	15 l	(40 l/h)



STEP 5

recycl. (loop I) (22 l/h)	11 l
recycl. (loop II) (75 l/h)	12 l
recycl. (loop III) (70 l/h)	11 l

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 94/00024

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C13D 3/14, B01D 15/08
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: C13D, B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 5127957 (H. HEIKKILA ET AL), 7 July 1992 (07.07.92) --	1-15
Y	US, A, 5102553 (M. M. KEARNEY ET AL), 7 April 1992 (07.04.92) --	1-15
A	GB, A, 2240053 (JAPAN ORGANO CO LTD), 24 July 1991 (24.07.91) -- --------	1-15

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
10 April 1994	16 -05- 1994
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Carolina Gomez Lagerlöf Telephone No. +46 8 782 25 00

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

16/04/94

International application No.
PCT/FI 94/00024

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 5127957	07/07/92	AU-A- 3500289	14/12/89
		CA-A- 1326850	08/02/94
		EP-A- 0345511	13/12/89
US-A- 5102553	07/04/92	AU-A- 4815690	10/07/90
		CA-A- 2005702	16/06/90
		EP-A- 0448633	02/10/91
		JP-T- 4502276	23/04/92
		US-A- 4990259	05/02/91
		WO-A- 9006796	28/06/90
		AU-A- 6908291	18/07/91
		WO-A- 9108815	27/06/91
GB-A- 2240053	24/07/91	DE-A- 4041414	27/06/91
		FR-A,B- 2656231	28/06/91
		JP-A- 4227804	17/08/92
		US-A- 5198120	30/03/93